FABRICATION AND CHARACTERISATION OF ALKALI TREATED NATURAL FIBRE REINFORCED POLYMER COMPOSITES

SUBMITTED BY

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CERTIFICATE

This is to certify that Sushree sangita mullick (roll no. 409ph2114) has submitted the thesis entitled "Fabrication and characterisation of natural fibre reinforced polymer composites" in partial fulfilment of the requirements for the award of master in science in the department of Physics , National Institute of Technology, Rourkela is an authentic work carried out under my supervision and guidance.

To the best of my knowledge, the matter contained in this thesis has not been submitted to elsewhere for the award of any degree.

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ABSTRACT

Coir fibre is treated with alkali for the improvement of the fibre properties. In order to measure all the effects on the coir fibre composite due to the alkali treatment various characterising tools such as XRD,SEM, FTIR and flexural tests are carried out. As a result of the treatment the improvement on the surface of the fibre is observed due to the reduction of the impurities, lignin content etc which is confirmed by SEM analysis an increase in fibre strength is also obtained which may be due to better fibre-matrix adhesion. A decrease in degree of crystallinity is observed during XRD analysis. However an increased physical and mechanical properties is observed due to the alkali treatment on the coir fibre.

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CHAPTER-1

INTRODUCTION

1.1 OVERVIEW OF COMPOSITES:

Processing of plastic composites using natural fibres as reinforcement has increased dramatically in recent years. The advantage of composite materials over conventional materials stem largely from their higher specific strength, stiffness and fatigue characteristics ,which enables structural designs to be more versatile. By definition these composite materials are engineered or naturally occurring materials made from two or more constituents materials with significantly different physical or chemical properties which remain separate and distinct at macroscopic and microscopic scale within the finished structure. These are materials that comprise strong load carrying material known as reinforcement imbedded in weaker material known as matrix. Reinforcement provides strength and rigidity, helping to support structural load. The matrix or binder (organic or inorganic) maintains the position and orientation of the reinforcement. The reinforcement may be particles, platelets or fibres and are usually added to improve mechanical property such as stiffness, strength and toughness of the matrix material. Long fibres that are oriented in the direction of loading offer the most efficient load transfer.

1.2 TYPES OF COMPOSITES:

In a broad way composite materials can be classified into three groups in the basis of matrix materials. They are:

1. Metal matrix composites (MMc)
2. Ceramic matrix composites (CMc)
3. Polymer matrix composites (PMc)

1.2.1 Metal matrix composites:

These composites have many advantages over monolithic metals like higher specific strength, higher specific modulus, better properties at elevated temperatures, and lower coefficient of thermal expansion. Due to these attributes metal matrix composites are under consideration for wide range of applications.

1.2.2 Ceramic matrix composites:

One of the main objectives in preparing ceramic matrix composites is to increase the toughness. Naturally it is hoped and also it is found that there is a concomitant improvement in strength and stiffness of ceramic matrix composites.

1.2.3 Polymer matrix composites:

Most commonly used matrix materials are polymeric. In general the mechanical properties of polymers are inadequate for many structural purposes. Generally their strength and stiffness are low compared to metals and ceramics. To overcome these difficulties other materials are reinforced with polymers. Secondly the polymer processing matrix composites need not involve high pressure and does not require high temperature. Also simple equipments are required for manufacturing polymer matrix composites. For this reason polymer matrix composites developed rapidly and have soon became popular for structural applications.

1.3. Natural fibre reinforced polymer composites:

The interest in natural fibre reinforced polymer composites is rapidly growing both in terms of their industrial applications and fundamental research. They are cheap, renewable, completely or partially recyclable, and biodegradable. Plants such as jute, flax, cotton, hemp, ramie, kenaf, bamboo etc. as well as wood, used from time immemorial as a source of lignocellulosic fibres, are more and more often applied as the reinforcement of composites. The natural fibre containing composites are more environmentally friendly, and are used in transportation such as automobiles, railway coaches, aerospace etc., military applications, building and construction industries (ceiling, panelling, and partition boards), packaging, consumer products etc. Natural fibre include those made from plants, animal and mineral sources. Naturally occurring fibres can be classified according to their origin.

1.4. Classification of natural fibre composites:

1.4.1. Animal fibre:

Animal fibres generally contain proteins; e.g. mohair , wool , silk , alpaca , angora. Animal hair contained fibres are the fibres taken from animals or hairy mammals . silk fibres are the fibres collected from the dried saliva of bugs or insects during the preparation of cocoons . Avian fibres are the fibres collected from birds, feathers.

1.4.2. Mineral fibres:

Mineral fibres are the naturally occurring fibres or slightly modified fibre procured from minerals. These may be of asbestos type, ceramic fibres or metal fibres.

1.4.3. Plant fibres:

Plant fibres are the fibres comprised of cellulose .e.g. ; cotton , jute , hemp, ramie , sisal , flax etc. These are further classified into the following categories:

i. Fruit fibre: these are extracted from the fruits of the plant. These are light and hairy and allow the winds to carry the seeds .e.g. coir i.e. coconut fibre.

ii. Bast fibre: these are found in the stems of the plant providing the plant its strength. Usually they run across the entire length of the stem are therefore very long.

iii. Leaf fibre: these are the fibres extracted from the leaves. These are rough and sturdy and form part of the plant's transportation system .e.g. sisal and agave.

In the beginning of the 20^{th} century wood or cotton fiber reinforced phenol or melamine formaldehyde resins were fabricated and used in electrical application for their non conductive and heat resistant properties. At present day natural fibre composites are mainly found in automotive and building industry and they are applied where load bearing capacity and dimensional stability under moist and high thermal conduction are of second order importance.

From a food and agricultural organisation survey, we have got the information about the fibres and countries of origin which is given in the following table.

Fibres	Countries of origin
Abaca	Malaysia,Uganda, phillipines , Bolivia.
Coir	India, srilanka, Philippines, Malaysia.
Flax	Borneo.
Hemp	Yugoslavia, china.
Jute	India, Egypt, Guyana, Jamaica , Ghana , Malawi , Sudan , Tanzania.
Kenaf	Iraq, Tanzania , Jamaica , south Africa ,cuba,togo.
Ramie	Handurus, mauritious.
Roselle	Borneo , Guyana , Malaysia , srilanka ,togo , Indonesia , Tanzania.
Sisal	East Africa , Bahamas , antique , Kenya , india.
Sun hemp	Nigeria ,Guyana , sierra leone , india.

Table 1.1 Fibers and countries of origin

Natural fibres such as jute , hemp , sisal , pineapple , abaca and coir have been studied as reinforcement and filler in composites. Taking into account the availability , growing attention is being paid to coconut fibre . The coconut husk is available in large quantities as residue from coconut production in many areas ,which is yielding the coarse coir fibre. coir is a ligno cellulosic natural fibre . It is a seed hair fibre obtained from the outer cell, or husk of the coconut. The total world coir production is 250000 tonnes. The coir fibre industries are particularly important in some areas of the developing world. Over 50% of the coir fibre produced annually throughout the world is consumed by in the countries of origin, mainly India. The other benefits of coir fibre are:

- ✓ 100% organic, biodegradable, renewable resources.
- ✓ High water and nutrition holding capacity, expands to hold up to 5 times its weight in water.
- ✓ Strong air porosity for excellent aeration.
- ✓ Absorption properties repel snails.
- ✓ pH 5.8 to 6.4.
- ✓ low electrical conductivity
- ✓ Water holding capacity up to 30%.
- ✓ low density of around 1.15 -1.33 kg/m^3

So like any other ligno-cellulosic fibers coir can act as a reinforcing material in the polymer composites. Surface of coir fiber can be modified chemically and physically with an aim to increase the adhesion between fiber and matrix. Chemical treatment of natural fiber often causes defibrillization , which also contributes to the increased reinforcing efficiency of the fibers in the composite. The removal of surface impurities which is a part of purification is advantageous for fiber–matrix adhesion as it facilitates both mechanical interlocking and the bonding reaction at the interface. In this regard, alkali treatment is a very suitable conventional technique.

Thus this report investigates the effects due to alkali treatment on the structure of coir fibre and also the mechanical properties of short coir fibre reinforced epoxy composite.

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CHAPTER -2

LITERATURE SURVEY

Here in this chapter the outlines of some of the recent reports published in literature on mechanical behaviour of natural fibre based polymer composites with special emphasis on coir fibre reinforced polymer composites.

2.1. On natural fiber reinforced composites

The mechanical properties of a natural fiber-reinforced composite depend on many parameters, such as fiber strength, modulus, fiber length, thier orientation, the fiber-matrix interfacial bond strength etc. A strong fiber-matrix interface bond is critical for high mechanical properties of composites. A good interfacial bond is required for effective stress transfer from the matrix to the fiber whereby maximum utilization of the fiber strength in the composite is achieved. Modification to the fiber also improves resistance to moisture-induced degradation of the interface and the composite properties. In addition, factors like processing conditions/techniques have significant influence on the mechanical properties of fiber reinforced composites. Mechanical properties of natural fibers like flax, coir, hemp, jute and sisal, are very good and may compete with glass fiber in specific strength and modulus. Numbers of investigations have been conducted on several types of natural fibers such as kenaf, hemp, sisal, flax, bamboo, and jute to study the effect of these fibers on the mechanical properties of composite materials. Mansur and Aziz studied bamboo-mesh reinforced cement composites, and found that these reinforcing materials could enhance the strength and toughness of the cement matrix, and increase its tensile, flexural, and impact strengths significantly. On the other hand, jute fabric-reinforced polyester composites were tested for the evaluation of mechanical properties and compared with wood composite, and it was found that the jute fiber composite has better strengths than wood composites. A pulp fibre reinforced thermoplastic composite was investigated and found to have a combination of stiffness increased by a factor of 5.2 and its strength increased by a factor of 2.3 relative to the virgin polymer. Information on the usage of banana fibers in reinforcing polymers is limited in the literature. In dynamic mechanical analysis, Laly*et al*. have investigated banana fiber reinforced polyester composites and found that the optimum content of banana fiber is 40%. Mechanical properties of banana–fiber–cement composites were investigated physically and mechanically by Corbiere-Nicollier *et al*. It was reported that kraft pulped banana fiber composite has good flexural strength. In addition, short banana fiber reinforced polyester composite was studied by Pothan*et al*.; the study concentrated on the effect of fiber length and fiber content. The maximum tensile strength was observed at 30 mm fiber length while maximum impact strength was observed at 40 mm fiber length. Incorporation of 40% untreated fibers provides a 20% increase in the tensile strength and a 34% increase in impact strength. Joseph *et al*. tested banana fiber and glass fiber with varying fiber length and fiber content as well. Luo and Netravali studied the tensile and flexural properties of the green composites with different pineapple fibre content and compared with the virgin resin. Sisal fibre is fairly coarse and inflexible. It has good strength, durability, ability to stretch, affinity for certain dyestuffs and resistance to deterioration in seawater. Sisal ropes and twines are widely used for marine, agricultural, shipping, and general industrial use. Belmeres et al. found that sisal, henequen, and palm fibre have very similar physical, chemical, and tensile properties. Cazaurang et al. carried out a systematic study on the properties of henequen fibre and pointed out that these fibres have mechanical properties suitable for reinforcing thermoplastic resins. Ahmed et al. carried out research work on filament wound cotton fibre reinforced for reinforcing high-density polyethylene (HDPE) resin. Khalid et al. also studied the use of cotton fibre reinforced epoxy composites along with glass fibre reinforced polymers. Fuad et al. investigated the new type woodbased filler derived from oil palm wood flour (OPWF) for bio-based thermoplastics composites by thermo gravimetric analysis and the results are very promising. Schneider and Karmaker developed composites using jute and kenaffibre and polypropylene resins and they reported that jute fibre provides better mechanical properties than kenaffibre. Sreekala et al. performed one of the pioneering studies on the mechanical performance of treated oil palm fiber-reinforced composites. They studied the tensile stress-stain behavior of composites having 40% by weight fiber loading. Isocyanine-, silane-, acrylated, latex coated and peroxide-treated composite withstood tensile stress to higher strain level.Isocyanate treated, silane treated, acrylated, acetylated and latex coated composites showed yielding and high extensibility. Tensile modulus of the composites at 2% elongation showed slight enhancement upon mercerization and permanganate treatment. The elongation at break of the composites with chemically modified fiber was attributed to the changes in the chemical structure and bondability of the fiber. Alkali treated (5%) sisal-polyester biocomposite showed about 22% increase in tensile strength. Ichazo et al. found that adding silane treated wood flour to PP produced a sustained increase in the tensile modulus and tensile strength of the composite. Joseph and Thomas studied the effect of chemical treatment on the tensile and dynamic mechanical properties of short sisal fiberreinforced low density polyethylene composites. It was observed that the CTDIC (cardanol derivative of toluene diisocyanate) treatment reduced the hydrophilic nature of the sisal fiber and enhanced the tensile properties of the sisal-LDPE composites. They found that peroxide and permanganate treated fiber-reinforced composites showed an enhancement in tensile properties. They concluded that with a suitable fiber surface treatment, the mechanical properties and dimensional stability of sisal-LDPE composites could be improved. Mohanty et al. studied the influence of different surface modifications of jute on the performance of the biocomposites. More than a 40% improvement in the tensile strength occurred as a result of reinforcement with alkali treated jute. Jute fiber content also affected the biocomposite performance and about 30% by weight of jute showed optimum properties of the biocomposites.

2.2. On coir fiber reinforced composites

Many aspects of the use of coir fibers as reinforcement in polymer–matrix composites are described in the literature. Coir is an abundant, versatile, renewable, cheap, and biodegradable lignocellulosic fiber used for making a wide variety of products. Coir has also been tested as a filler or a reinforcement in different composite materials. Furthermore, it represents an additional agro-industrial non-food feedstock (agro industrial and food industry waste) that should be considered as feedstock for the formulation of ecocompatible composite materials. Coconut coir is the most interesting products as it has the lowest thermal conductivity and bulk density. The addition of coconut coir reduced the thermal conductivity of the composite specimens and yielded a lightweight product. Development of composite materials for buildings using natural fiber as coconut coir with low thermal conductivity is an interesting alternative which would solve environment and energy concern. Geethamma et al. have studied the dynamic mechanical behaviour of natural rubber and its composites reinforced with short coir fibers.

Coir fiber–polyester composites were tested as helmets, as roofing and post-boxes. These composites, with coir loading ranging from 9 to 15 wt%, have a flexural strength of about 38 MPa. Coir–poly ester composites with untreated and treated coir fibers, and with fiber loading of 17 wt%, were tested in tension, flexure and notched Izod impact. The results obtained with the untreated fibers show clear signs of the presence of a weak interface long pulled-out fibers without any resin adhered to the fibers—and low mechanical properties were obtained. Although showing better mechanical performance, the composites with treated fibers present, however, only a moderate increase on the values of the mechanical properties analyzed. Alkali treatment is also reported for coir fibers. Treated fiber–polyester composites, with volume fraction ranging from 10% to 30%, show better properties than composites with untreated fibers, but the flexural strength of these composites was consistently lower than that of the bare matrix. A maximum value of 42.3MPa is reported against a value of 48.5MPa for the neat polyester. Acetylation of coir fibers increases the hydrophobic behaviour, increases the resistance to fungi attack and also increases the tensile strength of coir– polyester composites. However, the fiber loading has to be fairly high, 45 wt% or even higher, to attain a significant reinforcing effect when the composite is tested in tension. Moreover, even with high coir fiber loading fractions, there is no improvement in the flexural strength. From these results, it is apparent that the usual fiber treatments reported so far did not significantly change the mechanical performance of coir–polyester composites.

Although there are several reports in the literature which gives the idea of the mechanical behaviour of natural fiber reinforced polymer composites, very limited work has been done on mechanical behaviour of coir fiber reinforced epoxy composites. This research work has been undertaken, with an objective to explore the potential of coir fiber as a reinforcing material in polymer composites and to investigate its effect on the mechanical behaviour of the resulting composites.

2.3. Objectives of the Research Work:

The objectives of the project are outlined below.

- To develop a new class of natural fiber based polymer composites to explore the potential of coir fiber.
- Evaluation of mechanical properties such as: flexural strength, tensile modulus, micro-hardness, impact strength etc.

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CHAPTER-3

EXPERIMENTAL

3.1. Materials and methods:

The coir fibre is obtained from a local shop at Rourkela which has a dimension around 175 to 300 µm.Unmodified liquid epoxy resin based on Bisphenol A, of grade LY 556 along with hardener HY 951 is provided by B. Mukesh & Co., Kolkata, India. The density of the resin is 1.15 g/cc, whereas the hardener density is 0.97 g/cc.

3.2. Treatment of fiber :

The rough coir fibres are pre-washed with large amount of distilled water to remove the surface dirt present in the fibers and then put in an oven at 100°c for drying until it gains a constant weight. The mercerisation or alkali treatment is carried out by immersing the washed coir fibres (200gms) in a 10 %(w/v) sodium hydroxide aqueous solution (2lt) for 3hrs in a temperature of 70°c. It should be stirred occasionally and after that the fibres are taken out and washed in order to remove any absorbed alkali.

3.3. Fabrication of composite plate:

For the fabrication of the randomly oriented raw coir fiber-reinforced epoxy composite (RCFREC) and alkali coir fiber-reinforced epoxy composite (ACFREC) a handmade wooden mold is designed First. A releasing plastic sheet is spread over the wooden mold. Heavy duty silicon spray is applied to the plastic sheet for easy removal of the composite plate. The fibers are cut into 30 mm length and distributed uniformly at the bottom of the mold which is prepared before. Fifteen volume percentage of the fiber is used for the fabrication of the composite. Then a heavy load is applied on the mold containing the fibers for compression for around 24 hours. Then epoxy and hardener are mixed together on a weight percentage of 10:1 to form a matrix. The matrix is poured over the fibers in an evenly manner. Another releasing plastic sheet is spread over and then pressed and pushed down with an iron roller to avoid and eliminate any air bubble. Finally, load is given to it to remove excess matrix and left for curing at room temperature for 24 h. After that the desired composite plate can be obtained.

3.4. Characterization:

Chemical compositions of the raw and alkali treated coir fibres are investigated by the Perklin Elmer FTIR spectrometer spectrum RX-1 in the mid IR range i.e., from 400 to 4000 cm^{-1}.

The effect of treatment on the micro-molecular structures of coir fiber can be identified by wide angle X-ray diffraction (WAXD) spectra which are collected by PHILIPS PAN analytical PW1830 with Cu-K_{α} radiation from 10° to 45° with a scan speed of 0.04°/s. The crystal sizes of the fibers are determined by modified Scherer's formula whereas the degree of crystallinity is computed by comparing the areas under crystalline peak and amorphous curve.

In order to evaluate the flexural strength of the composites three point bending test is carried out by INSTRON1195. The randomly oriented RCFREC and ACFREC specimens are cut as per the ASTM D790 to measure the flexural strength. The samples taken for the flexural test is 160*30*5 mm which is cut by using a diamond cutter. Care has been taken for maintaining uniformity and homogeneity of the composite. The data reported here is the average of five successful tests.

Figure 3.1 specimen after the test

Also the fractured surfaces of the raw and alkali treated composites are examined by using SEM (JEOL JSM-6480 LV).

Figure 3.2 SEM Set up

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CHAPTER-4

RESULT AND DISCUSSION

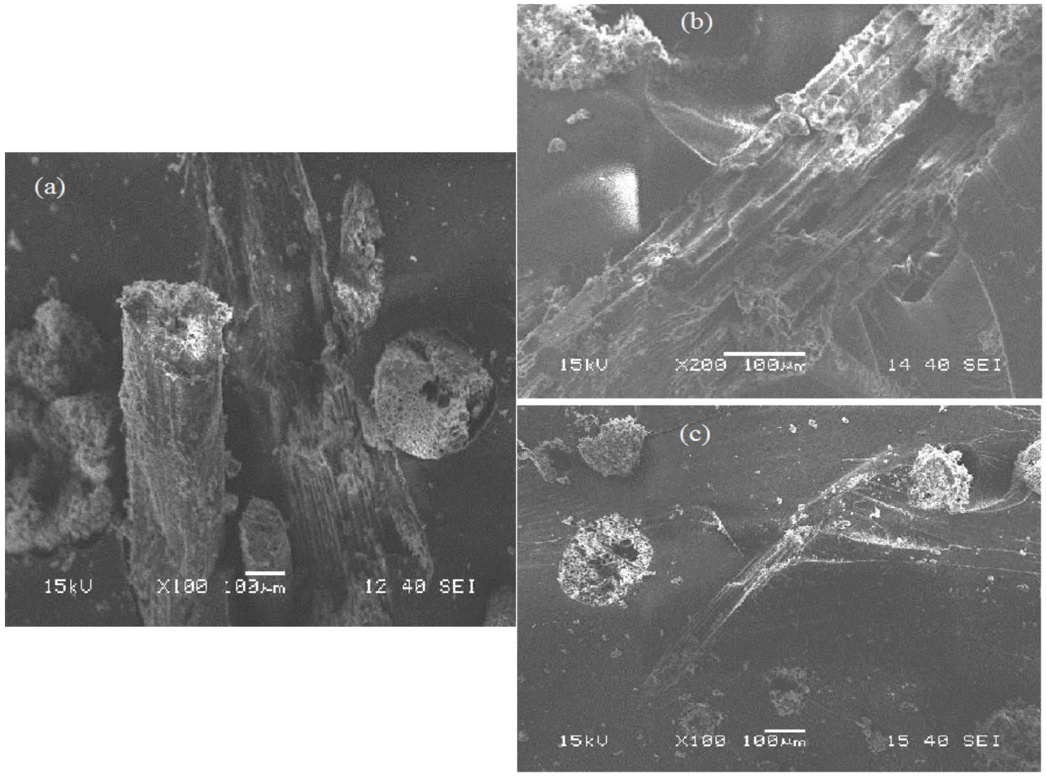

Figure 4.1. SEM analysis of raw coir fibre composite (a), alkali treated coir fibre composite (b),(c).

The SEM images of fractured surfaces of rough (a) and alkali treated coir fibre (b),(c) are shown in the figure. It is observed that the treatment has improved the surface roughness of the fibre as compared to the untreated fibre.This may be due to the reduction of fibre diameter which increases the aspect ratio ;thereby developing a rough surface. Also because of the removal of the surface impurities and cementing materials like lignin and hemicellulose it can be observed that separation of the ultimate cells has increased which leads to the increase in effective surface area of the fiber so it becomes more compatible with the matrix. From fig. (a) it is confirmed that the adhesion between the fibre and matrix is poor in the raw coir fibre composite, as there are gap around the fiber at the interface whereas, in the alkali treated composite, the fiber matrix adhesion has been increased which is shown by fiber breakage rather than fiber pullout in fig. (c).

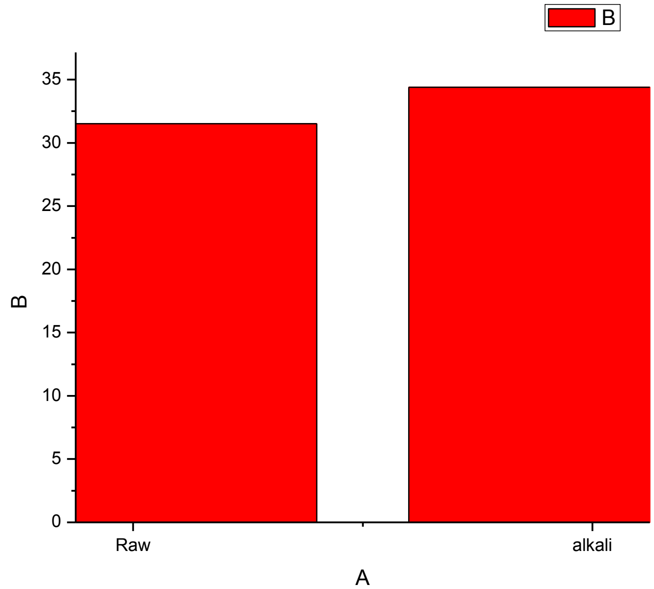

Figure 4.2 flexural strength of raw and alkali treated coir fibre composite

Figure shows that the flexural strength of both rough and alkali treated coir fibre . Here the strength of treated composite is found to be higher than the rough one. It is well known that for fiber reinforced composites, the interfacial zone plays a leading role in transferring the load between fiber and matrix which consequently affects the mechanical properties such as strength . As we know that flexural failure depends mainly on the fiber and matrix adhesion, the increased value of flexural strength in case of alkali treated composite may be due to the increase in effective surface area available for contact with the matrix (thesis).

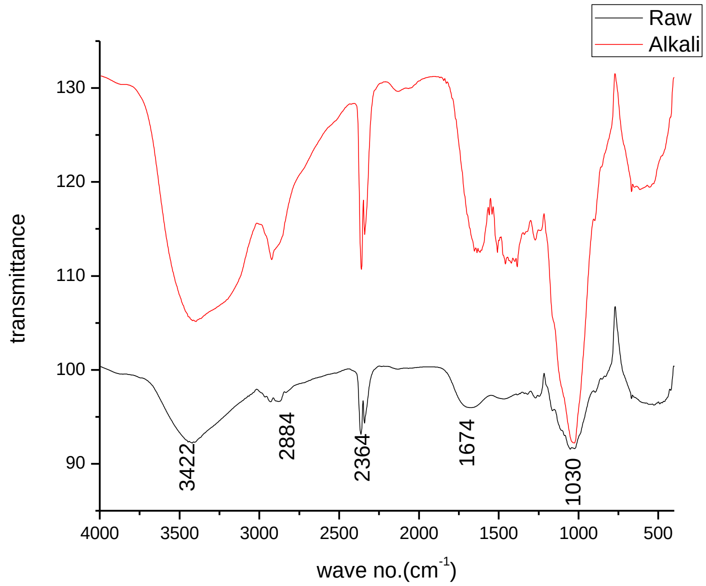

Figure 4.3. Image of FTIR analysis

The figure 4.3 above shows the FTIR data analysis for both raw and alkali coir fibres.

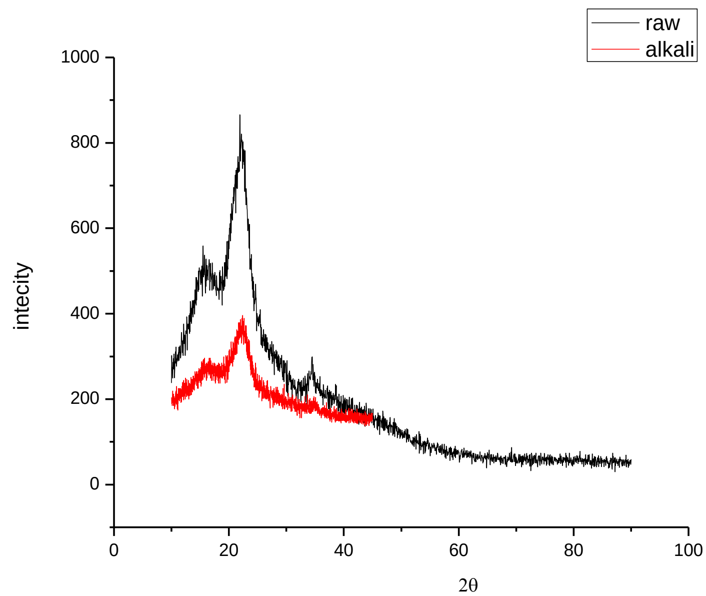

Figure 4.4. XRD analysis of raw and alkali treated fibre reinforced composites.

XRD patterns of both raw and alkali treated coir fibers are shown in Fig.4.4 . The full width at half maximum (FWHM) of the diffraction peaks of both raw and alkali treated coir samples are analyzed. The $K_{\alpha 2}$ components of the peaks are stripped from the data by Philips Xpert Highscore software. The diffractograms of both treated and raw coir fiber display a well-defined main peak around $2\Theta = 22.5°$, which is the characteristic of cellulose-I. By using the formula $\{1-(I_1/I_2)\}* 100$, we can calculate the degree of crystallinity for both the samples. It is found that the degree of crystallinity is found to be 73.033% and 50.3% for raw and alkali treated fibre respectively. This may be due to the swelling of the fibres due to the alkali treatment as this leads to the formation of new Na-cellulose-I lattice (Maya Jacob John).

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CHAPTER-5

CONCLUSION

In this project work coir fibre has been treated with alkali in order to analyse its modified properties. This study showed an increase in strength in case of treated fibre composite when flexural strength is studied . This may be due to the increased fibre matrix adhesion which is investigated by SEM analysis. This confirms the surface modified characteristics of the treated fibres which is due to the decrease in the amount of lignin , hemisolulose and impurity from the surface of the fibre.

This treatment leads to the fibrillation which causes the breaking down of the composite fibre bundles into smaller fibres. So a better adhesion between the fibre and matrix occurred. This treatment gave rise to a decrease in the degree of crystallinity which is proved by the XRD analysis of the raw and alkali treated fibres i.e. a swelling of fibre is occurred in the fibre due to the treatment.

So, this study concludes that alkali treatment can be done for improving the mechanical properties of the coir fibre composite.

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CHAPTER – 6

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